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(54) DISPERSIONS OF FATTY ACID MONO-/-DIGLYCERIDE MIXTURES

(71) We, CHEMISCHE FABRIK GRÜNAU GMBH a body corporate organised under the laws of Germany of 7918 Illertissen, Auer Strasse 100, Germany, do

hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to dispersions of fatty acid mono-/-diglyceride mixtures and to their use for the production of emulsions by the so-called cold method.

Cosmetic emulsions are normally produced by melting the product to be emulsified/emulsifier (oil phase) at a temperature of from about 60 to 70°C, heating the aqueous phase to the same temperature and combining the two phases at that temperature, both the water-to-oil and also the oil-to-water method being used.

The emulsion obtained after emulsification at about 60 to 70°C is cooled while stirring, optionally after homogenization, the temperature-sensitive ethereal oils being added during cold stirring at a temperature of from 35 to 45°C and the emulsion being cold stirred up to a temperature of from 20 to 30°C.

This method is primarily governed by the use of relatively high melting emulsifiers, such as waxes, fats and fatty alcohols, because these substances have to be converted into the liquid aggregate condition before emulsification.

In practice, it would appear that not only are these high-melting fractions heated to this temperature, the products to be emulsified which are liquid at temperatures as low as normal temperature, such as mineral oil, vegetable oil or isopropyl myristate, also have to be heated to this temperature. Since in general the water phase and oil phase are combined at the same temperature, the water is also heated to the high temperature. Before emulsification, a considerable quantity of energy has first to be applied in the form of heat and subsequently dissipated again by cooling after formation of the emulsion. Accordingly, this method is both time-consuming and energy-consuming.

In order to save energy, it has been proposed to stir cold water into the melt of the products to be emulsified and emulsifiers. However, this requires the use of special apparatus for producing an optimum emulsion.

In addition, it has been proposed to melt the relatively high melting fractions with some of the low-melting fractions and to add the rest of the low-melting fractions after the melt has been formed in order in this way to reduce the temperature of the oil phase, and subsequently to emulsify cold water into the temperature-reduced oil phase.

As used herein, the cold method of emulsification (cold emulsification) is the combination of the product to be emulsified, emulsifier and water for emulsion formation in the absence of heat, i.e. the combination of these components at temperatures of the order of 20°C.

Cold emulsification is possible in cases where the products to be emulsified and emulsifiers are liquid at normal temperature and can be combined with water at that temperature to form an emulsion. The fact that the products to be emulsified and the emulsifiers have to be liquid at normal temperature seriously restricts the choice of recipes from which cosmetic emulsions can be produced. Problems of consistency arise in emulsions produced in this way because in many cases waxes or other relatively high melting fractions are required for thickening pur-

poses. These thickeners have to be melted before emulsification to ensure that they are present in molten form during emulsification.

Hitherto it has only been possible to carry out cold emulsification on a limited scale, i.e. for the production of certain thinly liquid emulsions.

Cold emulsification would afford significant advantages, including

1. The saving of heat,
2. The saving of mechanical energy,
3. The saving of time,
4. A reduction in throughput,
5. The careful treatment of temperature-sensitive ingredients such as for example
   a) heat-sensitive oils
   b) vitamins
   c) ethereal oils.

Most attempts to carry out cold emulsification on a commercial scale have hitherto failed on account of the relatively high melting thickeners which have to be co-processed, and the problem of cold emulsification would be solved if it were possible for the thickening components to be able to be processed in cold form.

The present invention relates to a dispersion of a fatty acid mono-/-diglyceride mixture, characterised by a content of

a) from 10 to 60% by weight of a fatty acid mono-/-diglyceride mixture, of which the fatty acid monoglyceride content amounts to from 20 to 80 % by weight and of which the fatty acid diglyceride content amounts to from 75 to 15 % by weight,

b) from 1 to 20 % by weight of an anion-active surfactant comprising
   aa) alkali metal or alkylol ammonium salts of fatty acids with 10 to 20 carbon atoms, or
   bb) alkali metal or alkylol ammonium salts of fatty alcohol sulphates with 8 to 16 carbon atoms, or
   cc) alkali metal or alkylol ammonium salts of fatty alcohol polyglycol ether sulphates with 10 to 18 carbon atoms in the fatty alcohol radical and 2 to 5 oxyethylene units, or
   dd) alkali metal or alkylol ammonium salts of primary and/or secondary esters of phosphoric acid with alcohols which contain from 10 to 18 carbon atoms and which are optionally substituted in the two-position by a hydroxyl group, or
   ee) alkali metal or alkylol ammonium salts of fatty acid monoglycerides with 12 to 20 carbon atoms in the fatty acid radical esterified with tartaric or citric acid, and

c) 20 to 80% by weight of water.

Surprisingly, the dispersions according to the invention are able both to adjust the consistency of cosmetic emulsions and also to act as emulsifier for the quantities subsequently to be introduced of oils and ethereal oils which are liquid at normal temperature. It is particularly important that the dispersions according to the invention are present in a form which enables both oil and also water to be taken up.

Accordingly, the invention also provides a process for the production of an emulsion by the cold method as hereinbefore defined wherein one of the starting materials for the emulsion is a dispersion as described above.

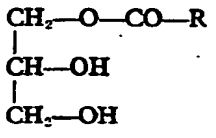
Fatty acid mono- and -diglycerides are monofunctional and difunctional esters of the trihydric alcohol, glycerin, and fatty acids. They may be produced by the direct esterification of glycerin with fatty acids or by the glycerinolysis of fats. Unfortunately, mixtures of monoglycerides, diglycerides and small quantities of triglycerides, free glycerin and free fatty acids, rather than pure products are obtained in both reactions. For example, the standard commercial-grade 50 % mono-/-diglycerides are mixtures consisting of about 40 to 50 % by weight of monoglyceride, about 30 to 40 % by weight of diglyceride and small quantities of triglyceride and glycerin.

The introduction of these fatty acid mono-/-diglyceride mixtures into water, even at temperatures above their melting point, does not result in the formation of an emulsion. However, mixtures of this kind may be converted into dispersions by means of suitable auxiliary emulsifiers. These dispersions vary in their physical/chemical behaviour, according to the auxiliary emulsifier used.

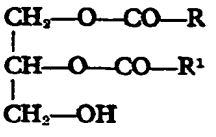
The dispersions according to the invention may be prepared by heating from 10 to 60 % by weight of a fatty acid mono-/-diglyceride mixture, of which the fatty acid monoglyceride content amounts to from 20 to 80 % by weight and its fatty acid diglyceride content to from 75 to 15 % by weight, together with 20 to 80 % by weight of water and 1 to 20 % by weight of the anion-active surfactant acting as auxiliary emulsifier to a temperature above the melting point of the glyceride mixture, i.e. to between 70 and 100°C, intensively stirring the melt and allowing the emulsion formed to cool while stirring to a temperature around room temperature.

The dispersions thus obtained are stable to ageing. They may be used for the production of cosmetic emulsions by the cold method either immediately or after storage for several months. It is possible to prepare both lotions and also solid creams, depending upon the quantity in which the dispersions are used.

Preferred fatty acid mono-/-diglyceride mixtures are mixtures of fatty acid monoglycerides corresponding to the general formula



and fatty acid diglycerides corresponding to the general formula



in which R and R^1 each represent the same or different, saturated or unsaturated, straight-chain or branched hydrocarbon radicals with 9 to 25 carbon atoms, optionally substituted by hydroxyl groups. It is particularly preferred to use mixtures of monoglycerides and diglycerides of the type for which, in the above formulae, R and R^1 each contain from 15 to 17 carbon atoms.

It is also of particular advantage for the proportion of fatty acid monoglyceride in the fatty acid mono-/-diglyceride mixtures to amount to from 30 to 60 % by weight and the proportion of fatty acid diglyceride to from 65 to 35 % by weight.

The anion-active surfactants acting as auxiliary emulsifier are preferably used in the form of salts of sodium, potassium or of mono-, di- or tri-alkylolamines of which the alkylol groups contain 2 or 3 carbon atoms.

Suitable salts are derived from;

aa) saturated or unsaturated, straight-chain or branched fatty acids with 10 to 20 carbon atoms; examples are corresponding salts of stearic acid, palmitic acid, oleic acid or mixtures thereof; or

bb) acid sulphuric acid esters of saturated or unsaturated, straight-chain or branched fatty alcohols with 8 to 16 carbon atoms; examples are lauryl sulphates, myristyl sulphates or mixtures thereof; or

cc) acid sulphuric acid esters of condensation products of saturated or unsaturated, straight-chain or branched fatty alcohols with 2 to 5 moles of ethylene oxide; examples are lauryl diethylene glycol sulphates, lauryl triethylene glycol sulphates or mixtures thereof; or

dd) primary and/or secondary esters of orthophosphoric acid with saturated or unsaturated, straight-chain or branched alcohols which contain from 10 to 18 carbon atoms and which are optionally substituted in the 2-position by a hydroxyl group; examples are cetyl phosphates (reaction product of cetyl alcohol and orthophosphoric acid in a molar ratio of 1:1), or phosphates of C_{14}—C_{16}-alcohols substituted in the 2-position by a hydroxyl group (reaction products of a C_{14}—C_{16} - α - epoxide mixture with orthophosphoric acid in a molar ratio of from 2:1 to 2.5:1); or

ee) fatty acid monoglycerides esterified with tartaric or citric acid, of which the fatty acid radicals are saturated or unsaturated, linear or branched and contain from 12 to 20 carbon atoms, for example the reaction product of glycerol monostearate with citric acid in a molar ratio of 1:1.

The invention is illustrated by the following Examples:

EXAMPLE 1

a) Preparation of a dispersion according to the invention:

25 parts of a fatty acid mono-/-diglyceride mixture, obtained by the glycerinolysis of hardened beef tallow, with a monoglyceride content of about 50 % by weight and a diglyceride content of about 45 % by weight, are heated with
5 parts of sodium lauryl sulphate and
70 parts of water

to a temperature of 70°C. On completion of melting, a dispersion is formed by intensive stirring, being cold-stirred up to 30°C. The dispersion thus obtained may be used for cold emulsification either immediately or after standing for several months.

b) Use of the dispersion prepared in accordance with a) for producing an emusion by the cold method:

50 parts of the dispersion prepared in accordance with a),
5 parts of 2-octyl decanol,
5 parts of isopropyl myristate,
5 parts of caprylic/capric acid triglyceride,
5 parts of paraffin oil,
5 parts of glycerol,
20 parts of demineralised water

are mixed at room temperature in a vessel equipped with contra-rotating stirrers. Mixing is over when a uniform cream has been formed. In order to increase stability in storage, it may be advisable subsequently to homogenise the cream.

The cream thus obtained is comparable in appearance, stability and cosmetic properties with a cream produced in the usual way by hot emulsification.

EXAMPLE 2

a) Preparation of a dispersion according to the invention:

40 parts of a fatty acid mono-/-diglyceride mixture, obtained by the glycerinolysis of hardened lard, with a mono-glyceride content of approximately 50 % by

weight and a diglyceride content of approximately 45 % by weight,
10 parts of a reaction product of a C_{14}—C_{16}-α-epoxide mixture with orthophosphoric acid in a molar ratio of 2:1, pH-value of the reaction product adjusted with triethanolamine to pH 6—7, and
50 parts of water

are processed to form a dispersion in the same way as described in Example 1a).

b) The use of the dispersion prepared in accordance with a) for the production of an emulsion by the cold method:

50 parts of the dispersion prepared in accordance with a),
7 parts of isopropyl myristate,
7 parts of paraffin oil,
8 parts of 1,2-propylene glycol,
25 parts of demineralised water,

are cold mixed to form an emulsion in the same way as described in Example 1b).

EXAMPLE 3

a) Preparation of a dispersion according to the invention:

45 parts of a fatty acid mono-/-diglyceride mixture, obtained by the glycerinolysis of hardened palm oil, with a monoglyceride content of approximately 40 % by weight and a diglyceride content of approximately 45 % by weight,
15 parts of cetyl phosphate diethanolamine salt and
40 parts of water

are processed to form a dispersion in the same way as described in Example 1a).

b) The use of the dispersion prepared in accordance with a) for the production of an emulsion by the cold method:

35 parts of the dispersion prepared in accordance with a),
6 parts of olive oil,
7 parts of 2-octyl dodecanol,
4 parts of isopropyl myristate,
4 parts of sorbitol,
15 parts of demineralised water,

are cold mixed to form an emulsion in the same way as described in Example 1b).

EXAMPLE 4

a) Preparation of a dispersion according to the invention:

25 parts of a fatty acid mono-/-diglyceride mixture, obtained by transesterifying hardened cottonseed oil, with a monoglyceride content of approximately 45 % by weight and a diglyceride content of approximately 50 % by weight,
5 parts of sodium stearate and
70 parts of water

are processed to form a dispersion in the same way as described in Example 1a).

b) The use of the dispersion prepared in accordance with a) for producing an emulsion by the cold method:

50 parts of the dispersion prepared in accordance with a),
8 parts of 2-octyl decanol,
8 parts of caprylic/capric acid triglyceride,
6 parts of sorbitol,
15 parts of demineralised water

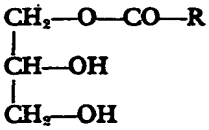
are cold mixed to form an emulsion in the same way as described in Example 1b).

WHAT WE CLAIM IS:—

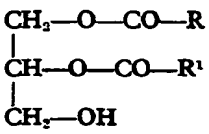
1. A dispersion of a fatty acid mono-/-diglyceride mixture, characterised by a content of

a) from 10 to 60 % by weight of a fatty acid mono-/-diglyceride mixture, of which the fatty acid monoglyceride content amounts to from 20 to 80 % by weight and of which the fatty acid diglyceride content amounts to from 75 to 15 % by weight,
b) from 1 to 20 % by weight of an anion-active surfactant from the group comprising
  aa) alkali metal or alkylol ammonium salts of fatty acids with 10 to 20 carbon atoms, or
  bb) alkali metal or alkylol ammonium salts of fatty alcohol sulphates with 8 to 16 carbon atoms, or
  cc) alkali metal or alkylol ammonium salts of fatty alcohol polyglycol ether sulphates with 10 to 18 carbon atoms in the fatty alcohol radical and 2 to 5 oxyethylene units, or
  dd) alkali metal or alkylol ammonium salts of primary and/or secondary esters of orthophosphoric acid with alcohols which contain from 10 to 18 carbon atoms and which are optionally substituted in the two-position by a hydroxyl group, or
  ee) alkali metal or alkylol ammonium salts of fatty acid monoglycerides with 12 to 20 carbon atoms in the fatty acid radical esterified with tartaric or citric acid, and
c) 20 to 80 % by weight of water.

2. A dispersion as claimed in Claim 1, wherein component a) is a mixture of fatty acid monoglycerides corresponding to the general formula



and fatty acid diglycerides corresponding to the general formula



in which R and R^1 each represent the same or different, saturated or unsaturated, straight-chain or branched hydrocarbon radicals with 9 to 25 carbon atoms optionally substituted by hydroxyl groups.

3. A dispersion as claimed in Claim 2, wherein R and R^1 each contain from 15 to 17 carbon atoms.

4. A dispersion as claimed in any of Claims 1 to 3, wherein the proportion of fatty acid monoglyceride in component a) amounts to from 30 to 60 % by weight and the proportion of fatty acid diglyceride to from 65 to 35 % by weight.

5. A dispersion of a fatty acid mono-/-diglyceride mixture substantially as described with particular reference to any of the Examples.

6. A process for the production of an emulsion by the cold method as hereinbefore defined wherein one component of the starting material for the emulsion is a dispersion as claimed in any of Claims 1 to 5.

7. A process for the production of an emulsion substantially as described with particular reference to any of the Examples.

8. An emulsion when prepared by a process as claimed in Claim 6 or 7.

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